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(21) International Application Number: PCT/US97/17601 (22) International Filing Date: 29 September 1997 (29.09.97) (30) Priority Data: 08/721,967 27 September 1996 (27.09.96) US (71) Applicant (for all designated States except US): NUCLEAR FILTER TECHNOLOGY, INC. [US/US]; Suite R, 741 Corporate Circle, Golden, CO 80401 (US). (72) Inventor; and (75) Inventor/Applicant (for US only): WICKLAND, Terry, J. [US/US]; 23722 Pondview, Golden, CO 80401 (US). (74) Agents: MOSES, John, R. et al.; Millen, White, Zelano & Branigan, P.C., Suite 1400, Arlington Courthouse Plaza I, 2200 Clarendon Boulevard, Arlington, VA 22201 (US).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: METHOD OF FABRICATING AND DEVICES EMPLOYING VENTS (57) Abstract <p>Vents for containers include a perforated substrate portion (20) of polymer material over which is thermally bonded a gas permeable membrane (30). The thermal bond (31) is direct and adhesive-free so that there is no adhesive available for chemical attachment by materials within the containers (58) and no opportunity for an adhesive to interfere with or block perforations through the substrate.</p> <div data-bbox="893 1113 1412 1470"> </div>		

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METHOD OF FABRICATING AND DEVICES EMPLOYING VENTS

Field of the Invention

5 The present invention is directed to vents, methods of fabricating vents and devices employing vents. More particularly, the present invention is related to vents which prohibit passage of liquids therethrough and allow passage of gases, wherein the vents are of particular interest with respect to venting containers having fluids therein which are hazardous.

Background of the Invention

10 Materials stored in containers frequently have both a liquid phase and a gas phase. With materials such as hazardous materials, it is frequently desirable to allow gas phases to vent while retaining liquid phases in the container.

15 An example of such hazardous materials is plutonium in aqueous hydrochloric acid or nitric acid solutions. As pipes and tanks containing these solutions are drained, they are generally drained into 1 gallon, high density, polyethylene bottles with polypropylene caps for interim storage until permanent solidification or immobilization may be conducted. The solutions contain as much as 140 g/l of plutonium which has the radiolytic effect of generating hydrogen gas. Hydrogen gas in the bottles accelerates embrittlement of the bottles which, over time, can lead to
20 failure of the bottles. Since plutonium is considered to be highly carcinogenic, leakage of plutonium into the environment surrounding the bottles poses a danger which

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5 must be avoided. Typically, venting of these bottles is through a GOR-TEX® element in the polypropylene cap. GOR-TEX® is impervious to liquids but pervious to gases. It has proven difficult to select an adhesive for securing the GOR-TEX® material to the caps because many adhesives tend to degrade in the presence of aqueous hydrochloric and nitric acid solutions containing plutonium.

10 This concern arises with respect to other structures such as the filter assembly disclosed in U.S. Patent 4,957,518 in which a GOR-TEX®, liquid permeable, but gas impermeable, barrier is employed. In this patent, the GOR-TEX® barrier may be held in place either mechanically or by an adhesive. Mechanical retention relies on clamping the GOR-TEX® barrier between two surfaces. Clamping can be unreliable. The other approach is, of course, to use adhesive, but, as previously stated, adhesive may be attacked by the material stored in the containers. In addition, there are fabrication problems which arise when using adhesive. This is because if a perforated substrate is used to support the GOR-TEX® barrier, the adhesive may flow over into and seal at least some or not all of the perforations.

15 In view of the aforementioned problems, as well as other problems, there is a need for new and improved vent structures; a need for a method for fabricating such structures; and a need for vent configurations employing the advantages offered by the improved vents.

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Summary of the Invention

25 It is a feature of the present invention to provide new and improved vents which are gas permeable and liquid impermeable; to provide a new and improved method of making such vents, and to provide new and improved structures utilizing the advantages of such vents.

In view of the aforementioned features, and other features, the present invention is directed to a lamination comprising polytetrafluoroethylene (PTFE) which is thermally bonded to a stiff, perforated substrate of resinous material.

30 A method of making a composite structure including a perforated substrate of resinous material and a layer of polytetrafluoroethylene (PTFE) comprises thermally

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bonding the layer of PTFE to the perforated substrate.

In a more specific aspect, the perforated substrate is porous and, in still a further aspect of the invention, the perforated substrate has discrete passages therethrough.

5 In a configuration for employing the previously described vent in accordance with the present invention, the vent is fabricated in a closure wherein the closure is a bottle cap or where the enclosure is an assembly for venting flexible containers such as bags.

10 In still a further aspect of the invention and further illustrative of the breadth of the invention, a vent configured in accordance with the principles of the present invention is used housings, such as the housings for battery cover electrical transformers to vent potentially explosive gases from the housings.

Brief Description of the Drawings

15 Various other features and attendant advantages of the present invention will be more fully appreciated as the same becomes better understood when considered in conjunction with the accompanying drawings, in which like reference characters designate the same or similar parts throughout the several views, and wherein:

20 **Figure 1** is a perspective view showing a polymer material substrate and a layer of polytetrafluoroethylene (PTFE);

Figure 2 is a side elevation showing the PTFE layer being thermally bonded to the perforated substrate;

Figure 3 is a side elevation showing gas or vapors passing through the lamination of Figure 2 and liquids being repelled by the lamination;

25 **Figure 4** is a side elevation of a bottle closure or cap utilizing the principles of the present invention as shown in Figure 3;

Figure 5 is a bottom view of the closure or cap;

Figure 6 is a top view of the closure or cap of Figures 4 and 5;

30 **Figure 7** is a side view of a container utilizing the cap of Figures 4-6 which employs the vent of Figure 3, fabricated in accordance with the methods of Figures

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1 and 2;

Figure 8 is a side elevation of a vent assembly utilizing the vent of Figure 3;

Figure 9 is a side, perspective view of a bag employing the vent assembly of Figure 8;

5 Figure 10 is a side elevation of a housing for an electrical transformer employing a vent such as the vent of Figure 3;

Figure 11 is a perspective view showing a electric iron which is used to thermally stake a layer of PTFE over a perforated portion of a large container cap;

10 Figure 12 is a view similar to Figure 11, but showing the electric iron applying heat to the PTFE layer;

Figure 13 is a side elevation showing the top portion of the cap of Figures 11 and 12 with a perforated cover snap fitted over the PTFE layer;

Figure 14 is a bottom view looking inside of a small cap comprising another embodiment of a cap in accordance with the present invention; and

15 Figure 15 is a side elevation of the cap of Figure 14.

Detailed Description

Referring now to Figure 1, there is shown a substrate 20 of a polymer material such as polyurethane, polypropylene, polyethylene, fiber glass reinforced polyethylene or other polymer materials. The substrate 20 is perforated by at least one discrete through hole 22 or by an array 24 of discrete through holes. Alternatively, the perforated substrate 20 is in the form of a stiff porous structure without discrete through holes, wherein fluids can migrate from a first side 26 to a second side 28 of the stiff substrate. In a preferred embodiment of the invention, the substrate 20 is stiff as opposed to flexible and the polymer material is a unitary mass rather than a stranded fabric, the substrate 20 preferably having the configuration of a plate.

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As is shown in Figure 2, a layer of polytetrafluoroethylene (PTFE), in the form of a membrane 30 such as one of the membranes identified by the trademark GOR-TEX® and disclosed in U.S. Patent No. 3,953,566, is thermally bonded to the

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substrate 20. GOR-TEX® membranes are available from G.L. Gore and Associate, Inc., Elkton, MD., and are discussed in U.S. Patent 5,264,276, issued November 23, 1993, incorporated herein by reference.

5 In thermal bonding, sufficient heat and pressure (indicated by arrows 31) are applied to the substrate 20 and the membrane 30 to cause thermal adhesion without unduly restricting the diameters of the discrete holes 22 or the hydrophobic characteristics of membrane 30, wherein the resulting lamination 32 is permeable to gas and vapor 33, but impermeable to liquid 34 (as is seen in Figure 3). In bonding a membrane 30 to a polypropylene substrate 20, low pressure is applied where the
10 GOR-TEX® membrane is rested against the substrate with slight pressure to insure engagement. The bonding temperature is in the range of about 150°C to about 200°C and is applied for about 0.5 to 15 minutes.

By thermally bonding the GOR-TEX® membrane 30 to the perforated substrate 20, no adhesive is required at the interface 35 of the GOR-TEX® membrane
15 30 and perforated substrate 20. Accordingly, there is no additional adhesive which can be degraded by whatever substance is present in the liquid repelled by the GOR-TEX® membrane. Moreover, the possibility is removed of closing the openings of the discrete through holes 22 by adhesive flowing from areas 36 adjacent the holes into interference with the holes. This difficulty can occur either during application
20 of an adhesive or upon pressing the membrane 30 into engagement with the first surface 26 of the substrate 20.

Referring now to Figures 4-6, there is shown a closure such as a bottle cap 40 employing the principles of the present invention. The bottle cap 40 is made up of a relatively stiff polymer material such as polypropylene and includes top 42 and
25 a cylindrical rim 44 having threads 46 for tight engagement with the neck of a bottle. The top 42 has an array 47 of discrete holes 48 therethrough. Heat bonded to the inner surface 49 of the top 42 is a GOR-TEX® membrane 52 and surrounding the GOR-TEX® membrane 52 is an annular lip 54.

As is seen in Figure 7, the cap 40 is threaded onto the neck 56 of a
30 polyethylene container 58 which contains material therein in the liquid

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phase 34 and the gas phase 33. The GOR-TEX® membrane 52 allows the gas phase 33 to vent or leave the container 58 while preventing the liquid phase 34 from leaving the container. Moreover, the GOR-TEX® membrane 52 prevents liquid from outside the container 58 from entering the container while allowing the relatively free exchange of air, or another surrounding gas, into and out of the container 58.

Referring now to Figure 8, there is shown another vent configuration 70 of the present invention. The vent configuration 70 has the general configuration of the vent assembly disclosed in U.S. Patent 4,957,518, issued September 18, 1990, and incorporated herein by reference. Vent assembly 70 is used to vent a bag 72 which contains radioactive waste materials or other hazardous waste materials. The vent assembly generally comprises a stiff but resilient base 74 having discrete holes 75 therein and a stiff but resilient cover 76 having discrete holes 77 therein. The cover 76 snaps into engagement with a rim on the walls of a chamber 78 containing a disk 79 of filter materials such as carbon-carbon filter material. In order to render the vent 78 gas permeable and liquid impermeable, a GOR-TEX® membrane 80 is interposed between the interior 81 of the bag 72 and the surrounding environment 82.

In accordance with one embodiment of the present invention, the GOR-TEX® membrane 80 is heat bonded to the bottom surface 83 of the cap 76 to cover the discrete holes 77. In still another approach, a GOR-TEX® membrane 97 is bonded to the base 74 between the filter element 79 and the base. In an alternative embodiment, a GOR-TEX® membrane 90 is thermally bonded to the bottom surface 92 of the base 74. In still another approach, the GOR-TEX® membrane 94 is thermally bonded to the upper surface 96 of the cover 76 in order to overlie the discrete through holes 77. In various embodiments and configurations, the GOR-TEX® membranes 80, 90 and 94 may all be employed, only the membranes 90 and 92 may be used or just the membranes 80 and 94 may be used. In any of the embodiments, an advantage of having at least one of the membranes 80, 90 and 94 thermally bonded to the base 74 or cover 76 is that the membranes are bonded in areas 98 between the discrete through holes 75 or areas 99 between the through holes 77. Such bonding over the entire interface substantially enhances the mechanical

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strength of the vent assembly. This is accomplished without depositing an adhesive in the areas 98 and 99 between the discrete through holes 77 and 75 which adhesive might interfere with or cover the openings of the holes.

Referring now to Figure 10, there is shown another embodiment of the invention, wherein a battery cover transformer housing 100 of a polypropylene material reinforced with fiberglass has a perforated area 102 with discrete holes 104 therethrough, which perforated area is covered by a GOR-TEX® membrane 106 that is thermally bonded to the perforated area. Accordingly, excessive pressure buildup within the transformer housing 100 due to generation of dangerous gases 108 is avoided while retaining dielectric liquid 110 within the transformer housing.

Referring now to Figures 11-13, there is shown a preferred method of heat bonding a GOR-TEX® membrane 200 to the inside surface 202 of the top 204 of a screw-on cap 206 in order to cover perforations 208. As is best seen in Figure 13, the inside surface 202 of the cap top 204 includes a first rib 209. The first rib 209 is surrounded by a second rib 210 which creates an annular channel 212 around the perforations 208. Preferably, the perforations 208 are in a raised area 214 of the top 204 so that there is a slight air gap 216 between the GOR-TEX® layer 200 and the inside surface 218 of the top 204 of the cap.

The GOR-TEX® layer 200 has a peripheral portion 220 which is secured in the annular trough 210 by heat staking with an iron 225 (Figs. 11 and 12). The iron 225 includes a head 228 that has a projecting rim 230 with an annular edge 232 which corresponds in diameter to the annular trough 210. As is seen in Figure 12, when the annular edge 232 of the heated head 228 is pressed against the peripheral portion 220 of the GOR-TEX® layer 200, it fuses with the polypropylene to form a unitary, sealed seam which is impenetrable by liquid or vapor.

A perforated cover 250 with perforations 252 therethrough is then snap-fitted over the GOR-TEX® layer 200 to protect the GOR-TEX® layer and to prevent touching of the GOR-TEX® layer by fingers which could damage the layer or deposit oil or some other contaminant on the layer. The perforated cover 250 has an annular side flange 254 which snaps beneath an annular shoulder 256 which is unitary with

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the top 204 of the cover.

Referring now to Figures 14 and 15, there is shown a smaller diameter version 206' of the cap 206 of Figures 11-13. In this version, similar primed reference numerals identify structure similar to the structure in Figures 11-13. The important similar feature is the GOR-TEX® membrane 200' that is thermally bonded in the circular channel 210 by the annular head 230 of an iron 225. In the embodiment of Figures 14 and 15, a cover such as the cover 250 is not employed because the cap is relatively small. It is, however, within the scope of this invention to utilize a perforated cover, such as the perforated cover 250, shown in Figure 13.

Without further elaboration, it is believed that one skilled in the art can, using the preceding description, utilize the present invention to its fullest extent. The following preferred specific embodiment is, therefore, to be construed as merely illustrative, and not limitative of the remainder of the disclosure in any way whatsoever.

EXAMPLE

Draining process pipes and tanks at nuclear facilities that contain as much as 140 g/l of plutonium in aqueous hydrochloric and nitric acid solutions provides a challenge for interim storage. As the pipes and tanks are drained into poly bottles
5 for interim storage until permanent solidification or immobilization may be conducted, the solutions are stored in 1 gallon, high density, polyethylene bottles with polypropylene caps. It has been observed that radiolytic effects, specifically, hydrogen gas generates pressures the bottles which accelerates embrittlement of the bottles.

10 In accordance with the present invention, hydrogen gas is safely vented from the bottles through a GOR-TEX® membrane which is permeable to vapors, yet impermeable to liquids. The membranes are mounted in pressure equalization caps which provides gas diffusion, yet retain liquids in the bottles in the event that the bottles capsize. The GOR-TEX® membranes are thermally bonded to the inside of
15 the cap in accordance with the present invention and provide caps which are physically durable and chemically resistant to the severe acid conditions of the liquid waste in the bottles.

It has been found that GOR-TEX® membranes thermally sealed to the polypropylene caps tolerate acidic vapors and withstand liquid head pressure of about
20 0.92 psi after 40 days of exposure to the acids within the bottles.

Compatibility tests performed show that the pressure equalization caps which had been exposed to 50% 6N nitric acid with 50% 6N hydrochloride acid withstood a 26 inch water column leak test with no visible degradation or change in the GOR-TEX® membrane. The only change in the cap is a yellowish/brownish discolorization
25 thereof.

When the caps were exposed to hydrochloric acid, no sign of degradation occurred and, when leak testing using a 20 inch water column, the cap proved leak-proof for 18 hours. Exposing the caps to nitric acid also showed no signs of degradation and proved leak-proof using a 26 inch water column for over 23 hours.

30 The pressure equalization vents comprised of GOR-TEX® thermally bonded to the

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interior of polyurethane caps on polyethylene bottles containing plutonium in acid solutions increases the use for life of the bottles. The GOR-TEX® membranes which are permeable to gases, yet impermeable to liquids, allow hydrogen to diffuse at a rate well above the TRUPACT-II minimum requirement of $1.9 \text{ E-6 mole/mol frac/sec}$. Moreover, such GOR-TEX® pressure equalization vents, thermally bonded to the caps, assure ambient pressure within the polyethylene bottles.

EXAMPLE 2

Six pressure equalization caps were prepared by thermally fusing GOR-TEX® membrane to the interior of the polypropylene cap. The non-woven polyester backing was on the clean side, and the expanded PTFE faced the acid solution. The caps were affixed to a bottle containing the designated test acid mixture. After allowing the caps to vent the bottles for a minimum of 40 days, each was tested to water entry at a pressure of 0.92 PSI (26" water column). None of the caps showed adverse degradation after compatibility testing, and none experienced water entry at the specified pressure.

No adhesive will sufficiently bond between the PTFE and most other polymeric materials – especially in an adverse environment such as acid hydrochloric and nitric acids.

PRESSURIZED WATER LEAK TEST RESULTS - AFTER ACID EXPOSURE

SAMPLE I.D.	ACID COMPATIBILITY		0.93 PSI LIQUID PRESSURE LEAK TEST	WATER ENTRY
	Test Acid Makers	Duration		
001-42-2-2	6.0 N HNO ₃	92 days	122 hrs	None
001-42-2-4	6.0N HCL	90 days	18 hrs	None
001-45-3-1	HCL	42	9 hrs	None
001-45-3-2	HNO ₃	41	16.5 hrs	None
001-45-3-3	50 6.0 N HCL 50 6.0 N HNO ₃	41	8.5 hrs	None
001-45-3-4	50 6.0 N HNO ₃ 50% 6.0 N HCL	44	hrs	None

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The preceding examples can be repeated with similar success by substituting the generically or specifically described reactants and/or operating conditions of this invention for those used in the preceding examples.

5 From the foregoing description, one skilled in the art can easily ascertain the essential characteristics of this invention, and without departing from the spirit and scope thereof, can make various changes and modifications of the invention to adapt it to various usages and conditions.

WHAT IS CLAIMED IS:

1. A vent allowing passage of gas or vapor therethrough but preventing passage of liquid therethrough, the vent comprising:

a perforated substrate of polymer material; and

a layer of liquid impermeable, but gas permeable, material thermally bonded to the layer of perforated polymer material with an adhesive-free interface.

2. The vent of claim 1, wherein the polymer material is selected from the group consisting of fiber reinforced polypropylene polyurethane, polypropylene and polyethylene.

3. The vent of claim 2, wherein the layer of gas permeable, liquid impermeable material is polytetrafluoroethylene.

4. The vent of claim 1, wherein the layer of gas permeable, liquid impermeable material is polytetrafluoroethylene.

5. The vent of claim 1, wherein the polymer material of the substrate is a stiff, non-stranded material.

6. A vent for a container, said vent being permeable to gas or vapor but impermeable to liquid, the vent comprising:

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a stiff substrate of polymer material, the substrate having an array of discrete through holes therethrough in spaced relation to one another to define the areas therebetween;

a GOR-TEX® membrane thermally bonded to at least one side of the substrate with a thermal bond that includes bonding in the areas between the through holes, the thermal bond being an interface between the substrate and membrane which is direct and free of adhesive.

7. The vent of claim 6, wherein the polymer material of the substrate is selected from the group consisting of polyurethane, polypropylene, polyethylene and fiber reinforced polymers.

8. A vented container for containing acidic solutions of a radioactive material, the container comprising:

a bottle of a polymer material having an opening;

a closure for the opening, the closure being made of a polymer material and having an area including an array of spaced through holes therein; and

a membrane of GOR-TEX® material thermally bonded directly to the area of the closure having the array of through holes over an adhesive-free interface of the area and the GOR-TEX® membrane which is adhesive-free.

9. The vented container of claim 8, wherein the container body is polyethylene and the closure is of polypropylene.

10. The vented container of claim 9 further including the contents thereof, wherein the contents thereof comprises plutonium in an acidic solution.

11. The vented container of claim 10, wherein the acidic solution is a solution of aqueous hydrochloric acid.

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12. The vented container of claim 10, wherein the acidic solution is an aqueous solution of nitric acid.

13. A vent for venting gas through an opening in the wall of a container, the vent comprising:

- a base having a perforated portion with an array of discrete through holes therein;

- a cover having an area with an array of discrete through holes therein;

- a GOR-TEX® membrane thermally bonded to at least one of the areas having an array of discrete through holes therein over a direct interface between the GOR-TEX® membrane and the area, which direct interface is adhesive-free.

14. The vent of claim 13 further including a block of filter material disposed between the cover and the base, wherein the block of filter material is a barrier to the passage of solid particles through the vent, the GOR-TEX® membrane permitting passage of gas or vapor through the vent and preventing passage of liquid therethrough.

15. The vent of claim 14, wherein the base and cover clamp therebetween material of the container with which the vent is used.

16. A housing for an electrical transformer, wherein the housing contains fluid in a liquid phase and fluid in a gas phase, the housing comprising:

- an area in a wall of the housing having an array of discrete through holes therein, at least the area being comprised of a polymer material; and

- a GOR-TEX® membrane thermally bonded directly to the area having the array of through holes therethrough over substantially the entire area including portions of the area between the through holes, the thermal bond being over an interface which is adhesive-free.

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17. A method of fabricating a vent through a wall, the method comprising:
providing at least one hole through a substrate of polymer material;
thermally bonding a layer of GOR-TEX® material directly to the substrate
over the hole with an adhesive-free interface.

18. The method of claim 17, wherein a plurality of holes in an array within
a selected area are provided.

19. The method of claim 18, wherein the substrate is polypropylene and the
adhesive-free interface extends over the entire selected area.

20. The method of claim 17, wherein the substrate is a stiff pervious material
in a unified mass and the hole is one of many passages through the material.

21. The method of claim 17, wherein there are a plurality of holes through
the substrate of polymer material, and wherein the layer of GOR-TEX® material has
a peripheral portion surrounding the plurality of holes, the method further including
applying heat only to the peripheral portion to bond the peripheral portion to the
substrate of polymer material.

22. The vent of claim 6, further including an annular rib surrounding the
array of discrete through-holes, wherein the polytetrafluoroethylene membrane has
a peripheral portion which extends beyond the annular rib and wherein the
polytetrafluoroethylene membrane is bonded to the substrate of polymer material in
an area radially external to the annular rib.

23. The vent of claim 22 further including a second annular rib surrounding
the first annular rib to define a circular channel therebetween, the circular channel
receiving the peripheral portion of the polytetrafluoroethylene membrane which is
bonded to the substrate.

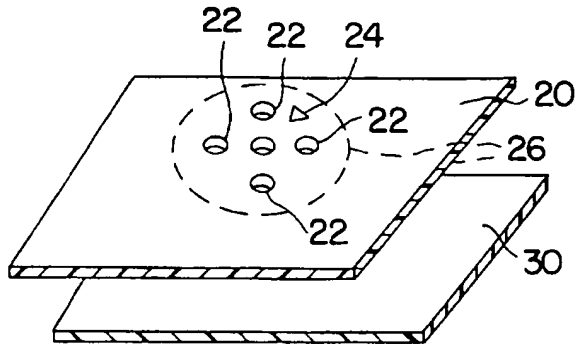


FIG. 1

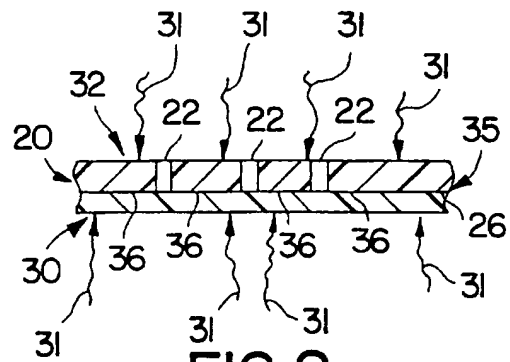


FIG. 2

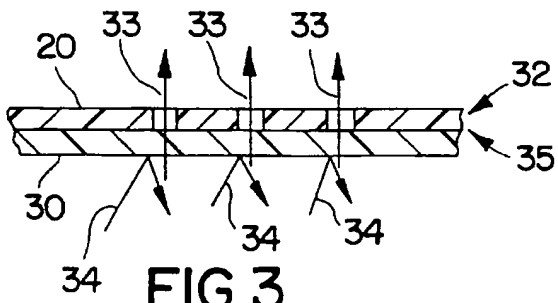


FIG. 3

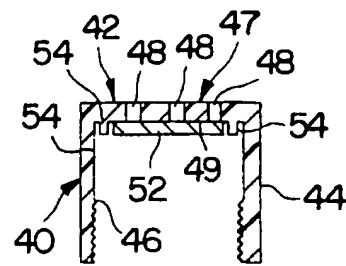


FIG. 4

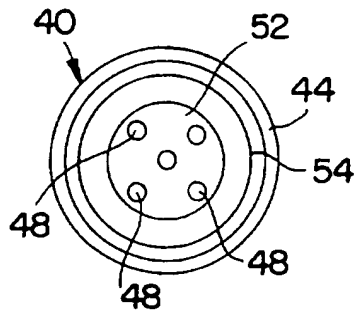


FIG. 5

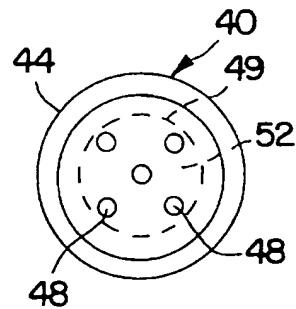


FIG. 6

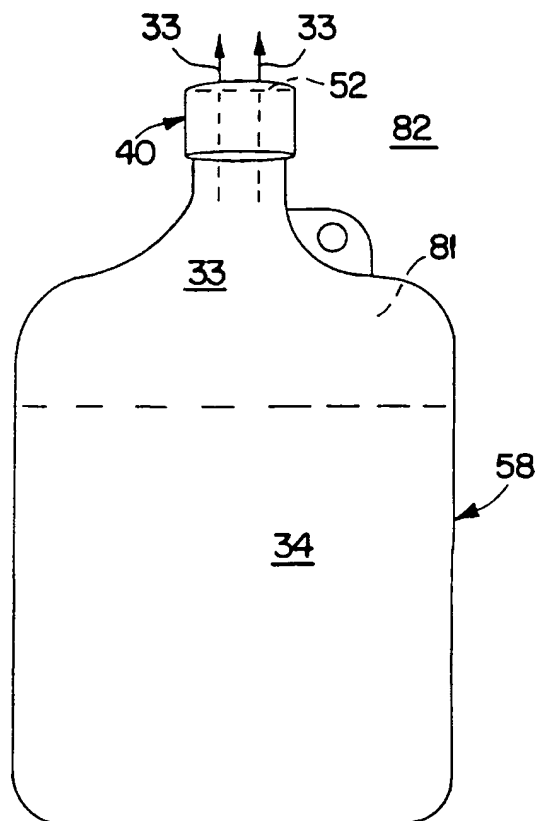


FIG. 7

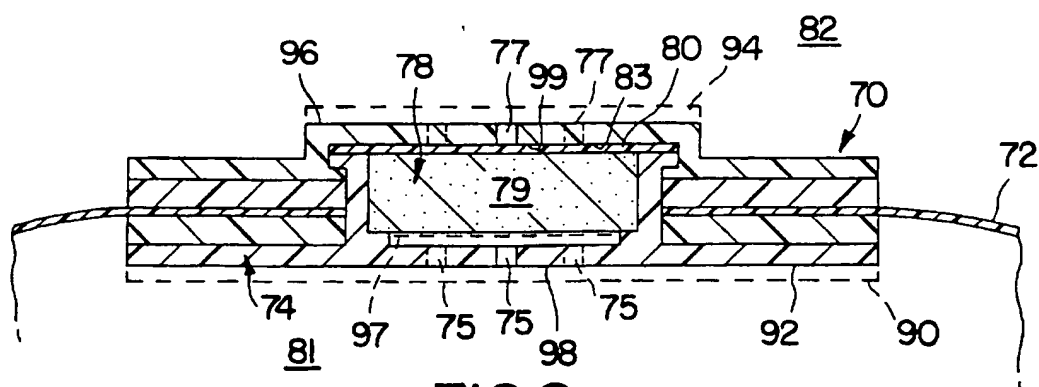


FIG.8

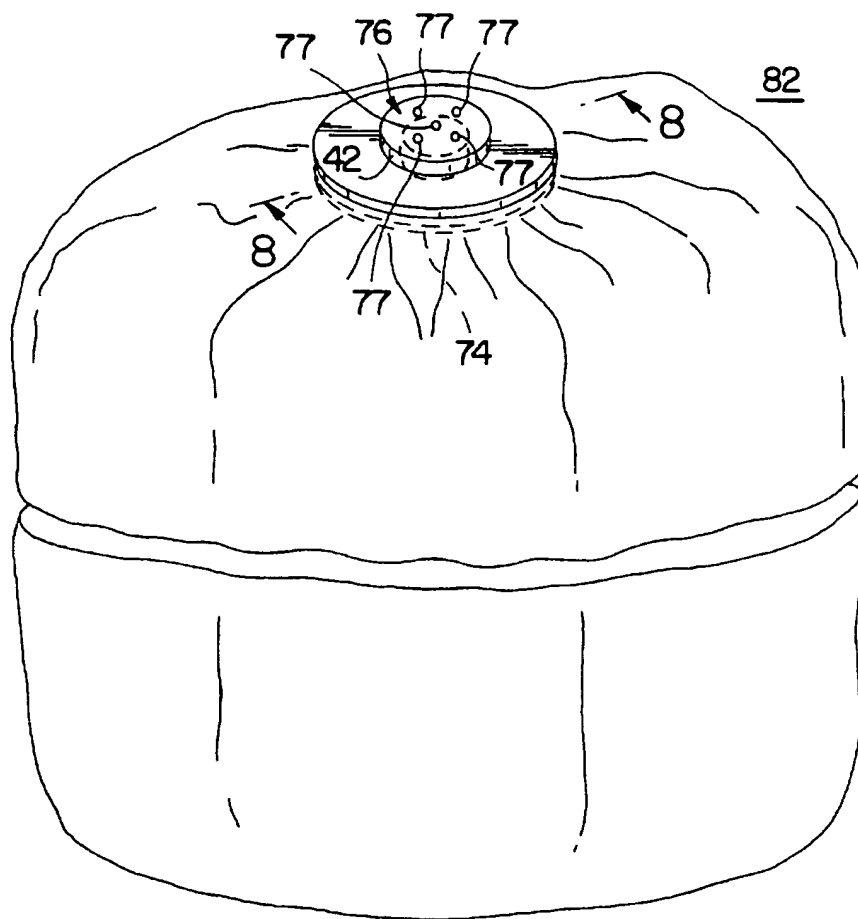


FIG. 9

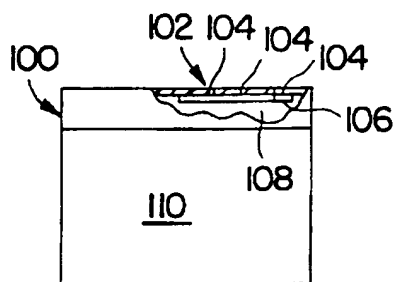
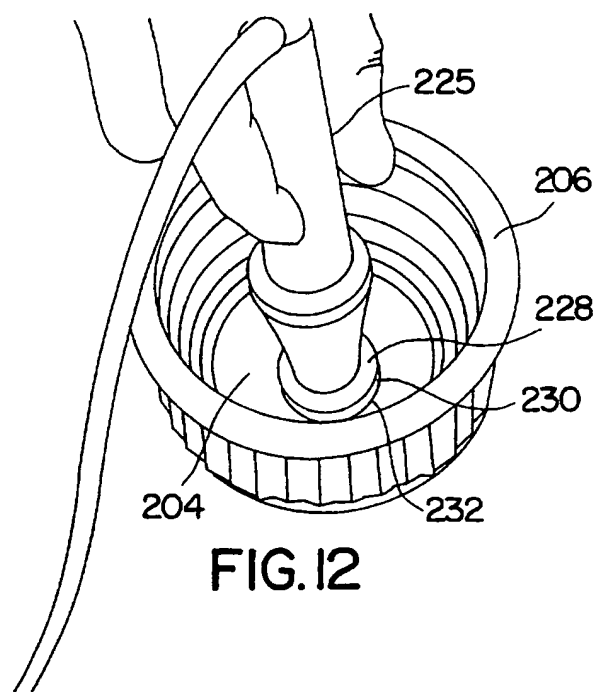
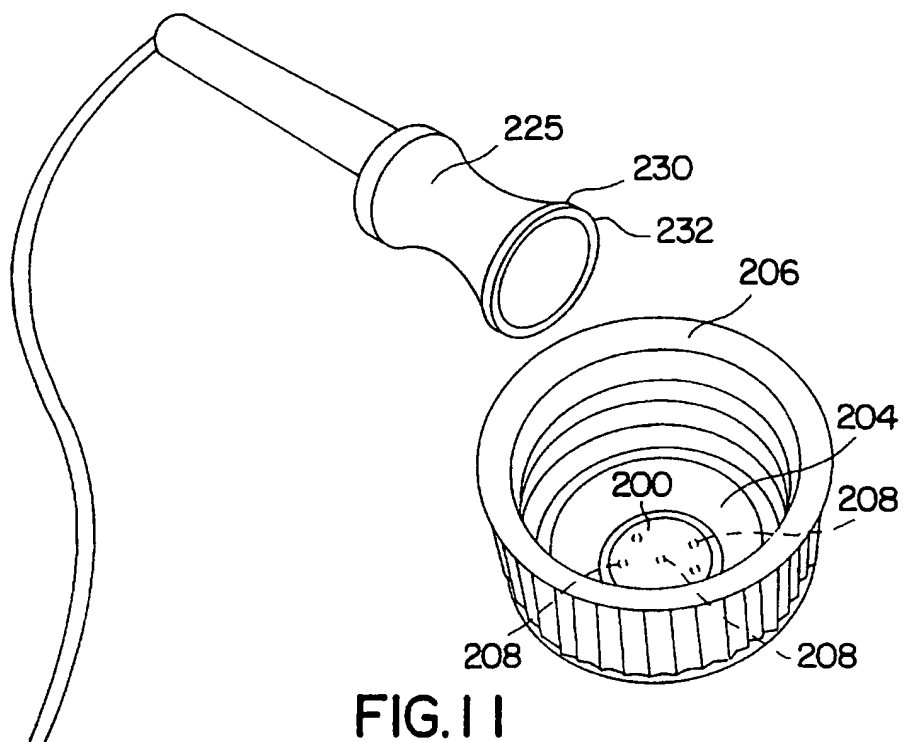


FIG. 10



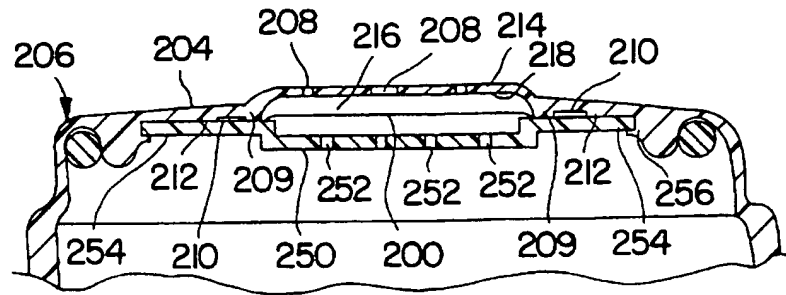


FIG. 13

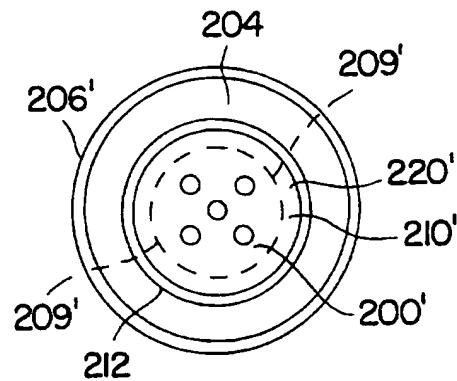


FIG. 14

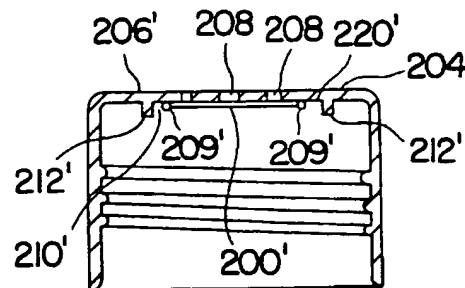


FIG. 15